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16. Abstract

A comparative experimental study on the application of various tests for the evaluation of the fire resistant property of plastic materials as presented. On the basis of the results obtained conclusions are drawn on the advantages and disadvantages of the methods used, justifying the option for a method considered as being of universal nature.

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EXPERIMENTS OF THE SELECTION OF A METHOD EVALUATING THE FIRE RESISTANCE OF SOME MATERIALS BASED ON MACROMOLECULAR COMPOUNDS

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Plastic materials are replacing to an increasing extent /175* traditional natural materials in the most different sectors. The advantages they present are however decreased somewhat because of their characteristics of their behavior with regard to fire.

Materials on the basis of macromolecular compounds can not be considered as totally noncombustible because being organic materials, they are destroyed in a fire lasting a long time. By improving the resistance to fire of macromolecular compounds the purpose is inparticular the self extinction of the material after the removal of the initiating external sources or the elimination of the possibility of rapid propagation of the fires.

Many fires have been indicated [reference 1] in cable systems of large electrical stations, telephone exchanges, industrial units and computer centers. Their frequency and gravity of these fires have caused a justified anxiety among planners, builders and the operating personnel of the plants, the more so as the common element of the fires listed was a massive participation of the use of conductors and cables insulated with PVC and neoprene, considered as being very refractory.

Besides the losses of material mentioned, thousands of human lives are lost every year in fires in buildings [reference 2], as a result of the combined effects of the flames, fumes and toxic gases arising, while materials based on macromolecular compounds are considered responsible to a great extent.

Up to now the method for characterizing the resistance to fire of macromolecular compounds did not make it possible to correlate reconditions of a fire and laboratory procedures. It is considered that the methods of investigation are of relative nature and can be

*Numbers in the margin indicate pagination in the foreign text.

used only to have a comparative evaluation of the behavior under fire of polymer materials.

The study of the fires registered so far show the need of starting some studies whose purpose would be primarily the prevention of fires, the control of the fire, suppression of the focus, the decrease of the amount of fumes and toxic gases produced by the fire.

Within the framework of fire prevention, an important rule is assigned to the use of materials with improved resistance to fire, as well as the establishment of methods of testing the resistance to fire which would secure the correspondence between the behavior of material in the laboratory and in conditions of a real fire [reference 3,4]. Sometimes keeping in mind the importance of knowing the resistance to fire of polymer materials, the products are exposed to a controlled fire on scale and in operating conditions [reference 1].

The principle by improving the resistance to fire we achieve autoextinction (self extinction) of the material after the removal of the initiating source or the nonignition of plastic material after a given period of initiation.

The treatment applied on a material to delay/prevent the propagation of flames is called fire retardants [reference 6]. Fire retardant agents can be incorporated in plastic materials as additive or as reactive chemical groups in basic polymers, in the polymerization process.

The fire retardant additive may be obtained by adding to polymer compounds an agent which in case of a fire would prevent the penetration of air into the combustion zone, forming a protective crust (phosphates, halogenated derivatives, borates, silicates) or layers of noncombustible gases (HCl, HBr, CO_2) [reference 7,8].

The choice of a fire retardant agent requires that we should take into consideration (apart from the problem of economy) the stage and phase (gas or condensate) in which the burn process must be delayed, the optimum quantity of fire retardant agent, the effect

TABLE 1

FLAMMABILITY TEST

/176

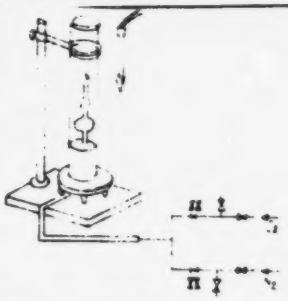
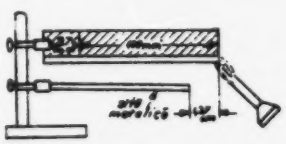
Method	Principle of the method	Determining characteristic	Evaluation of the behavior
ASTM-D	 The burning of the test bar in a measured mixture of oxygen and nitrogen in which the oxygen percentage is controlled to determine the minimum concentration required to maintain combustion.	The limiting index for oxygen, % speed of combustion, mm/min.	The ASTM does not provide a classification of the materials. Ishii[reference 11] classifies the material according to the need for oxygen for a combustion: <20% -easily flammable 20-25% -flammable 26-30% -repels the flame 31-50% - repels powerfully the flame >50% - nonflammable
ASTM-D 635	 The horizontal sample is exposed for a period of 30 seconds to the flame of a Bunsen burner and the combustion is followed up to the marking of 100 mm	Speed of combustion cm/min Length burnt, mm Time of extinction, s	Burns under this test does not burn in this test is self extinguished in this test

TABLE 1

FLAMMABILITY TEST

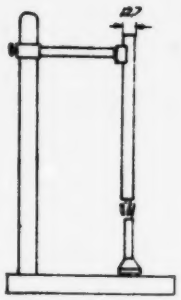
Method	Principle of the method	Determining characteristic	Evaluation of the behavior
UL-94 V			
	The samples oriented vertically are exposed to the flame of a Bunsen burner for a period of 10s. If the combustion begins in less than 30s, we ask for a second application of 10s. Over 305 mm, a layer of cotton is applied. The phenomenon of dropping is followed, the time of extinction and incandescence.	Class of Combustion: V-0	$-t_s$ (extinction time)<5s; the drops do not ignite the cotton; $-t_i$ (time of incandescence)<30s.
		V-1	$-t_s$<25s; the drops do not ignite the cotton; $-t_i$<60s.
		V-2	$-t_s$<25s; the drops ignite the cotton; $-t_i$<60s.

TABLE 2

/177

RESISTANCE TO FIRE OF COMPOUNDS ON THE BASIS OF POLYETHYLENE (PE), CHLORINATED POLYETHYLENE (PEC) AND ANTIMONY TRIOXIDE (Sb_2O_3)

Proba nr. a	ASTM-D 635		ASTM-D 2863	UL—94 V	
	t_p, s	b lungimea arsă, mm	c indicele-limită de oxigen, % (LOI)	d clasa de ardere	e Comportare în condiții similare unui incendiu în exploatare—galerii experimentale
1	234	38,9	26,9—25,6	f Nu corespunde	g Propagă incendiul
2	105	26,9	26,3—26,5	Nu corespunde	Propagă incendiul
3	211	39,3	25,8—26	Nu corespunde	Propagă incendiul
4	175	27,9	26,5	Nu corespunde	Propagă incendiul
5	8	20,7	29,03	V—O	h Nu propagă incendiul
6	6	16,7	27,9	V—O	Nu propagă incendiul
7	6	29,1	27,8	V—O	Nu propagă incendiul
8	3	29,1	27,8	V—O	Nu propagă incendiul
9	2	23,3	34,8	V—O	Nu propagă incendiul
10	3	22	26,5	V—O	g Propagă incendiul

Key: a. Sample number
b. length burned, mm
c. limiting indicator oxygen % (LOI)
d. combustion class
e. Behavior in similar conditions of fire under operation experimental gallery.
f. No correspondence
g. Propagates fire
h. Does not propagate fire

TABLE 3

LIMITING INDEX OF OXYGEN FOR COMPOUNDS ON THE BASIS OF PE i.d., PEC + Sb_2O_3

Content in PEC, %	0	5	10	15	20	30	40	50	75	100
Limiting index of oxygen, %	19,5	20,9	23,9	25,1	24,9	24,00	24,8	25,9	26,6	29
		20,7	22,8	24,4	24,7	24,00	23,4	25,3	26	28,9

TABLE 4

LIMITING INDEX OF OXYGEN FOR COMPOUNDS ON THE BASIS OF PE j.d., PEC + Sb_2O_3

Content in PEC, %	0	5	10	15	20	30	40	50	75	100
Limiting index of oxygen, %	19,5	21,1	24,17	25,6	25,6	25,9	26,7	26,7	26,96	29

[Typist's note: in the preceeding tables, decimals are indicated by commas.]

of the fire retardant agent on the physical and mechanical qualities of the compound (in general the resistance to shock, elongation, the resistance to traction under rupture, etc., deteriorate) and the toxicological conditions occurring at the time of handling the fire retardant compounds.

The effect of delaying the flame (inhibition) is attributed to a variety of compounds, which contain especially elements of two groups (V and VII) of the Periodic Table: N, P, As, Sb, Bi and heavier elements, F, Cl, Br, I [reference 8].

One of the systems used in fire retardant macromolecular compounds is the synergic system Cl-Sb_2O_3, because both of its technical performances and from the toxicological aspect. Antimony chloride and antimony oxychloride obtained at the time of combustion are assumed to form an effective protection crust, which is maintained over a long time in the combustion zone and contributes to the reduction of the concentration of free radicals capable of oxidation [reference 9] by transfer of halogen and formation of carbon inside the solid phase. As compared with the fire retardant agents which form protective layers of noncombustible gases, Sb_2O_3 by forming a crust, is of a less toxic nature.

The purpose of investigations carried out on materials with known composition is to compare the different methods of evaluation of resistance to fire, in view of choosing a laboratory test considered as being of representative nature as regards the behavior under fire of materials.

The known test used over a long period on a world wide level /177
for characterizing the resistance to fire of plastic material may may be grouped as follows:

1. According to needs:

--test of investigation from which comparable technical values are obtained;

--test of acceptance required of the contractor (supplier-beneficiary).

2. According to the source and time of initiation:

--test of low severity; the exposure is to a small amount of heat over a short time;

--test of maximum severity; it is exposed to a large amount of heat over a prolonged time.

3. According to characteristics of flammability [reference 7]:
--test to determine the possibility of ignition;
--test to determine the propagation of the flame front;
--test to determine the combustion heat;
--test to determine the evolution of smoke;
--test to determine the endurance of the fire;

The relative flammability varies considerably with the test used. Likewise a single test is insufficient, but for economical reasons it is impossible to imply all the tests for characterization purposes. Likewise the laboratory tests have an inherent deficiency, it is assumed that they cannot reproduce the effects of combustion heat, the presence of fire on a macroscale (conflagration) and that is the results can not be representative if they are applied in this context [reference 10]. For example it is possible that because of the temperatures resulting in a conflagration, the products which confer resistance to fire of the plastic materials should begin to volatilize and/or undergo thermal destruction, so that the remaining material no longer possesses the characteristic of resistance to fire.

Table 1 gives the methods used and considered as representative to determine the resistance to fire (easy inflammation and propagation of the flame front) of fire retarding polyethylenes. Other methods known (STAS-8790/71, ASTM D-2843 etc.) make it possible to determine other characteristics of flammability or are specific methods of a category of products (STAS 7015, ASTM-D 1692 etc.), such as sheets, expanded product etc.

Table 2 gives a series of characteristics of the resistance to fire for different compounds tested according to the methods indicated.

It may be considered that the samples which are in an /178
experimental gallery (in which the conditions of a real conflagration are simulated) do not propagate the fire if they have an oxygen index larger than 27. That is the limiting index of oxygen may

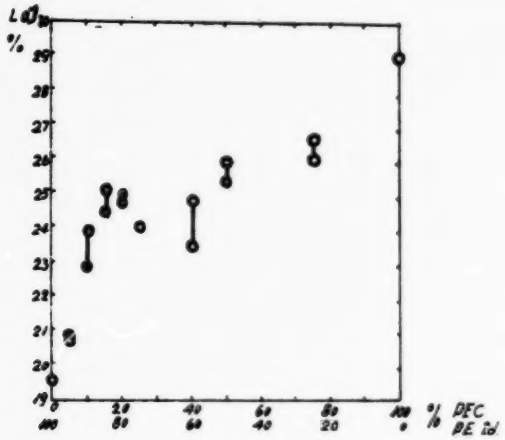


Fig. 1. Limiting index of oxygen as a function of the content in PEC of the compounds of PEC-PE i.d.

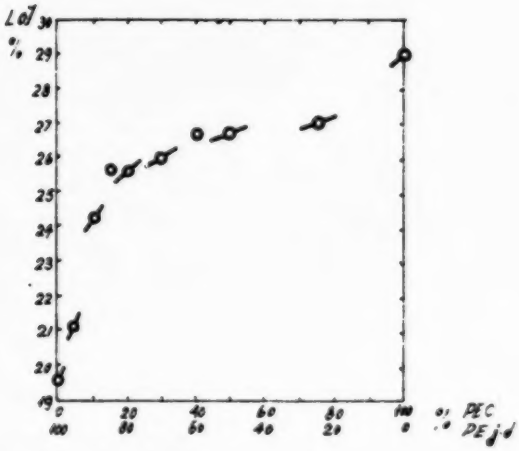


Fig. 2. Limiting index of oxygen as a function of the content in PEC of the compounds of PEC-PE j.d.

establish a correlation between the laboratory conditions and the effective conditions of a conflagration. The ASTM-635 method indicates (according to the table) the values which can not be correlated with the results obtained in the experimental gallery. Thus sample number 10 according to this method indicates values similar to the samples 5-9, though greater. Meanwhile sample 10 allowed the propagation of the conflagration, contrary to the values of the limiting index of oxygen (26.5 %), from which a conclusion may be drawn on the behavior of the material in conditions of conflagration.

Table 3 gives the values of the determinations carried out according to the method ASTM D-2863 (limiting index of oxygen) for high density polyethylene (PE i.d.) fire proofed with chlorinated polyethylene (PEC) in the presence of Sb_2O_3.

It is observed that the limiting index of oxygen increases with the increase of the contact of PEC in PE, that is it follows perfectly the variation of the chlorine content introduced in the compound (fig.1) up to a level of approximately 15% PEC in PE. Between 20 and 70% PEC in PE i.d., although the chlorine content in the compound increases considerably, the oxygen indices show

very close values, with lower increase. This phenomenon may be explained by the fact that Sb_2O_3 (maintained in constant amounts in all compounds) achieves synergism with the halogenated compound (PEC) within a certain optimum ratio.

Table 4 (fig. 2) presents values of the determination according to ASTM D-2863 for low density polyethylene (PE j.d.) fire proofed with PEC and Sb_2O_3.

It is observed that in the case of PE j.d. using the method of oxygen index, we find faithfully an increase of the resistance to fire with the increase of the chlorine content and the compound.

The comparative study of the methods analyzed (ASTM-D 635, UL-94 V, UL-94 V, and ASTM-D 2863) it was found that the most conclusive method to determine the resistance under fire (facility of ignition) of plastic material is the method of the limiting (critical) indices of oxygen. This method may be the basis for implementing the necessary correlation to establish the behavior of materials in real conflagration conditions with laboratory tests.

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